



THE FRANKLIN INSTITUTE • PHILADELPHIA 3, PA. • LOCust 4-3600
LABORATORIES FOR RESEARCH AND DEVELOPMENT

February 2, 1965

UNPUBLISHED PRELIMINARY DATA

N65-82208

Code rose
Case CR 60835

National Aeronautics and Space Administration
Washington 25, D.C.

Reference: Contract No. NASr-145

Subject: Quarterly Progress Report Q-B2028-9, covering the period
October 22, 1964 to January 21, 1965.

Gentlemen:

The research effort during this period has been concerned with: (i) a repeat of the bend fatigue test on a single crystal oriented for basal slip (ii) a repeat of the bend fatigue test on a single crystal oriented for prism slip (iii) an analysis of the loop vectors in the as-fatigued and recovered condition.

(i) Bend Fatigue Basal Slip Orientation

A second single crystal oriented for basal slip was fatigued by reverse bending. The load was increased in increments until fracture occurred at the fixed grip with a resolved shear-stress on the basal plane of 3460 psi. This stress level is approximately four times the resolved shear-stress for plastic flow on the basal plane measured in a simple tension; the previous bend crystal of this orientation fractured at a resolved shear-stress of 3124 psi. Examination of the surface by optical microscopy and electron microscopy of high resolution silicon monoxide replicas confirmed the operation of slip only on the basal plane. A layer $\sim 1\mu$ was removed from the bend surface by electro-polishing and the new surface replicated. Examination of the replicas revealed the presence of rows of etch-pits delineating the sites of the original surface basal slip structure. The procedure was repeated after removal of a further $\sim 3\mu$ from the surface; in this case, no evidence was found for the original slip structure.

B2028

These slip observations were confined to one of the two bend surfaces, the other having been protected by a layer of microstop. The crystal was sliced (spark-machining) into rectangular pieces suitable for transmission electron microscopy. These pieces are currently being annealed at temperatures up to $\sim 500^{\circ}\text{C}$, in order to study the recovery process in bulk fatigued beryllium. In this case, foils will be prepared from the protected surface layer following the annealing treatment.

(ii) Bend Fatigue Prism Slip Orientation

A second single crystal oriented for prism slip has been fatigued in simple bending. The discoloration on the upper and lower bend surfaces was again noted, the intensity of the discoloration increasing with increasing number of cycles. As in the case of the first prism slip crystal, the side faces (those approximately parallel to the $1/3 \langle \bar{2}110 \rangle$ slip vector) remain untarnished throughout the fatigue life of the crystal. The surface slip structure and dislocation substructure of this crystal is being examined.

(iii) Dislocation Loops and Burgers Vectors

A. Fatigued Condition: The available evidence strongly suggests that the loops formed during fatigue lie on the basal plane. This is true for both basal and prism slip orientations. Possible Burgers vectors associated with these loops are: the partial vectors $\frac{1}{2}[0001]$ (Frank dislocation) and $1/6 \langle 20\bar{2}3 \rangle$ (Frank-Shockley composite dislocation); and the perfect vectors $1/3 \langle 11\bar{2}3 \rangle$ and $1/3 \langle 11\bar{2}0 \rangle$. The diffraction contrast experiments have eliminated $\frac{1}{2}[0001]$ since the loops appear in contrast in foils having a basal plane orientation and using reflections from planes normal to the basal plane. Further, the $1/6 \langle 20\bar{2}3 \rangle$ partial vector is eliminated since the loops are extinct using reflections of the type $10\bar{1}0$. Thus, the loop vector is either $1/6 \langle 11\bar{2}3 \rangle$ or $1/3 \langle 11\bar{2}0 \rangle$ which means that in either case the loops do not give rise to a stacking fault. It is not possible to verify the absence or presence of fringes indicative of faulting in the inclined loops since the extinction distances in beryllium approach the total foil thickness. For example, the extinction distance is $\sim 1500 \text{ \AA}$ using the 0002 reflection. Further diffraction contrast experiments are necessary to differentiate between $1/6 \langle 11\bar{2}3 \rangle$ and $1/3 \langle 11\bar{2}0 \rangle$.

0002 151 0001

B. Recovered Condition: Extensive recovery was observed at ambient temperatures in foils prepared from bend fatigue crystals of both orientations. In the case of basal bend fatigue, the large dislocation loops appear to belong to one specific set of crystal planes. From their projected shapes, the loops could be either on the (0001) plane or on the ($\bar{2}$ 110) plane. The former appears to be more likely since it is difficult to envision why only one of the three possible {11 $\bar{2}$ 0} planes should be favored as the loop plane. The observation of a closed loop 'ghost' image after the disappearance of the actual loop is best explained in terms of a combination of climb and prismatic glide; this requires that the loops have a Burgers vector out of the plane of the loop. In the case of the prism orientation-bend fatigue crystals the projected shape of the loops observed after recovery, is consistent with their lying on the active (01 $\bar{1}$ 0) slip plane.


Since the extinction distances in beryllium are of the same order of magnitude as the foil thickness, fringes are not expected, even in large faulted loops, should these be present. However, the appearance of the loop image does give an indication of the nature of the loop. Thus, the small loops, probably on (10 $\bar{1}$ 0), constituting the recovered structure of the prism slip crystal show two distinct forms of image contrast, namely light or dark inside the loop. This could arise from differences in the depth of each inclined loop from the foil surfaces, provided the loops are faulted. In comparison, the absence of any variation of image intensity inside the large loops of the recovered structure of the basal slip crystal points to their being perfect. These loops are believed to lie on the (0001) plane. For a better understanding of the recovery mechanism, a unambiguous determination of the loop vectors for both crystal orientations is necessary. This is possible with the use of specific diffraction contrast conditions for image formation in the electron microscope.

C. Mechanism of Loop Formation: The occurrence of dislocation loops having a direction of elongation normal to each of the three possible $1/3 \langle 2110 \rangle$ vectors can be accounted for by the model of Johnston and Gilman (1) in which a moving screw dislocation becomes jogged by cross slip. Motion of the jog will then give rise to either rows of vacancies or interstitials, or of two edge segments of opposite sign, depending on the height of the jog. Whether or not the coalescence of point defects to form prismatic loops can occur will depend on the point defect mobility in the beryllium at the fatigue temperature (300°K). Insufficient data are available in the literature to allow for a quantitative assessment of the interstitial atom mobility. However, by comparison with other close packed metals, and taking into account the

relatively high Debye temperature (1160°K) it is estimated that interstitials are relatively mobile at ambient temperature. Thus, if trails of interstitials are formed at the small jogs these will coalesce to give interstitial loops. An estimate of the vacancy mobility can be made from a knowledge of the activation energy for self-diffusion in Beryllium (Lee et al (2), Naik et al (3)). This activation energy is approximately 1.7 eV, and if it is assumed that approximately one-half of this energy represents the activation energy for vacancy migration, the calculated jump frequency is sufficient to expect some coalescence of vacancy trails into vacancy loops at ambient temperatures.

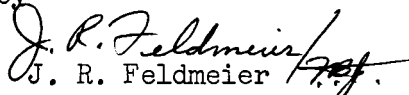
Larger jog heights of several atomic spacings give rise to trails of plus-minus edge dislocation dipoles. These may reduce their energy by pinching off to form small circular loops, or they may have their spacing reduced or be terminated by the segments of the original screw dislocation moving together again. This has been observed directly by Damiano and Herman (4), and there are several instances of this form of configuration in the present study.

The cause of the jogs is not completely clear. Although the glide dislocations intersect the grown in dislocation networks, the jogs formed in this way are glissile since the Burgers vectors of the stationary and moving dislocations are parallel and in the basal plane (4). It is possible that in a metal such as beryllium 'obstacles' are present and that these give rise to sessile jogs on moving screw dislocations.


H. Conrad
Technical Director


A. Lawley, Manager
Physical Metallurgy Laboratory

Approved by


J. R. Feldmeier
Director of Laboratories

REFERENCES

1. W. G. Johnston and J. J. Gilman, J. Appl. Phys. 31, 632, 1960.
2. C. H. Lee, J. B. Drew, G. E. Spangler, and M. Herman, Second Int. Conference on Beryllium, Met., Franklin Institute, Phila., Penna. AIME - to be published, 1965.
3. M. C. Naik, J. M. Dupouy, and Y. Adda, *ibid.*
4. V. V. Damiano, and M. Herman, Mat. Science Research, Vol. II, Plenum Press, New York, 1965.